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**PATENT COOPERATION TREATY (PCT)
TRAITÉ DE COOPÉRATION EN MATIÈRE DE BREVETS (PCT)**

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A ÉTÉ DÉPOSÉE, AINSI QUE DE TOUTES CORRECTIONS Y RELATIVES**

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Demande internationale n° }

International Filing Date
Date du dépôt international }

19 MARCH 2003

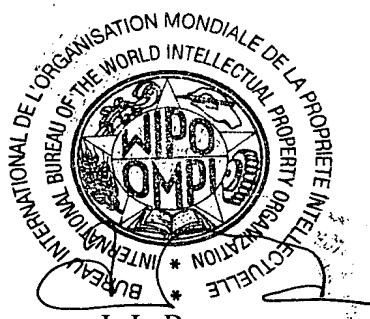
19.03.03

Geneva/Genève, 23 FEBRUARY 2004

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PCT REQUEST

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0	For receiving Office use only	
0-1	International Application No.	PCT / IB 03 / 01079
0-2	International Filing Date	19 MARCH 2003 (19.03.03)
0-3	Name of receiving Office and "PCT International Application"	INTERNATIONAL BUREAU OF WIPO PCT International Application
0-4	Form - PCT/RO/101 PCT Request	
0-4-1	Prepared using	PCT-EASY Version 2.92 (updated 01.01.2003)
0-5	Petition The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty	
0-6	Receiving Office (specified by the applicant)	International Bureau of the World Intellectual Property Organization (RO/IB)
0-7	Applicant's or agent's file reference	5870-PCT
I	Title of invention	ODORANT COMPOUNDS
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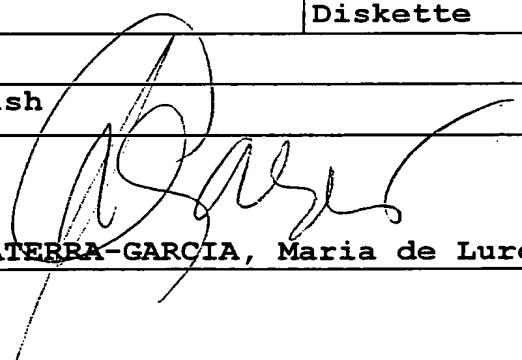
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IV-1	Agent or common representative; or address for correspondence The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: Name (LAST, First) Address:	agent SALVATERRA-GARCIA, Maria de Lurdes FIRMENICH SA 1, route des Jeunes P. O. Box 239 CH-1211 GENEVA 8 Switzerland +41 22 780 22 11 +41 22 780 33 38
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V	Designation of States	
V-1	Regional Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AP: GH GM KE LS MW MZ SD SL SZ TZ UG ZM ZW and any other State which is a Contracting State of the Harare Protocol and of the PCT EA: AM AZ BY KG KZ MD RU TJ TM and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT EP: AT BE BG CH&LI CY CZ DE DK EE ES FI FR GB GR HU IE IT LU MC NL PT SE SI SK TR and any other State which is a Contracting State of the European Patent Convention and of the PCT OA: BF BJ CF CG CI CM GA GN GQ GW ML MR NE SN TD TG and any other State which is a member State of OAPI and a Contracting State of the PCT
V-2	National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH&LI CN CO CR CU CZ DE DK DM DZ EC EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NI NO NZ OM PH PL PT RO RU SC SD SE SG SK SL TJ TM TN TR TT TZ UA UG US UZ VC VN YU ZA ZM ZW

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V-5	Precautionary Designation Statement In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation(s) of the State(s) indicated under item V-6 below. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit.		
V-6	Exclusion(s) from precautionary designations NONE		
VI	Priority claim NONE		
VII-1	International Searching Authority Chosen European Patent Office (EPO) (ISA/EP)		
VIII	Declarations	Number of declarations	
VIII-1	Declaration as to the identity of the inventor	-	
VIII-2	Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent	-	
VIII-3	Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application	-	
VIII-4	Declaration of inventorship (only for the purposes of the designation of the United States of America)	-	
VIII-5	Declaration as to non-prejudicial disclosures or exceptions to lack of novelty	-	
IX	Check list	number of sheets	electronic file(s) attached
IX-1	Request (including declaration sheets)	4	-
IX-2	Description	11	-
IX-3	Claims	2	-
IX-4	Abstract	1	EZABST00.TXT
IX-5	Drawings	0	-
IX-7	TOTAL	18	
	Accompanying items	paper document(s) attached	electronic file(s) attached
IX-8	Fee calculation sheet	✓	-
IX-17	PCT-EASY diskette	-	Diskette
IX-19	Figure of the drawings which should accompany the abstract		
IX-20	Language of filing of the international application	English	
X-1	Signature of applicant, agent or common representative		
X-1-1	Name (LAST, First)	SALVATERRA-GARCIA, Maria de Lurdes	

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10-1	Date of actual receipt of the purported international application	19 MARCH 2003 (19. 03. 03)
10-2	Drawings:	
10-2-1	Received	
10-2-2	Not received	
10-3	Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application	
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10-5	International Searching Authority	ISA/EP
10-6	Transmittal of search copy delayed until search fee is paid	

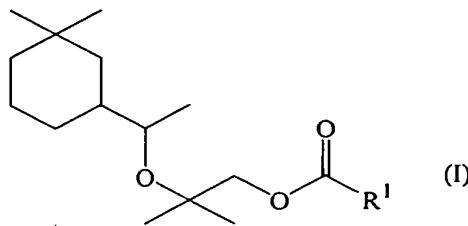
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11-1	Date of receipt of the record copy by the International Bureau
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ODORANT COMPOUNDS

Technical field

5 The present invention relates to the field of perfumery. More particularly, it concerns an unsaturated compound of formula (I)



10 as defined further below.

The present invention concerns also the use of said compound as perfuming ingredient as well as the compositions or articles associated with said compound.

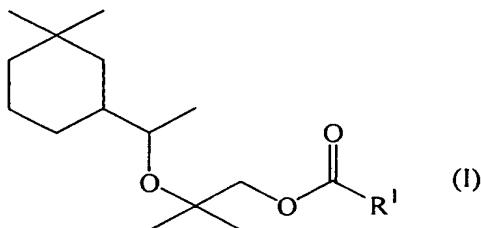
Prior art

15 To the best of our knowledge only one compound of formula (I) has been reported in the prior art, namely 2-[1-(3,3-dimethylcyclohexyl)ethoxy]-2-methylpropyl 2-methyl-2-butenoate. Said compound has been reported in EP 1262474 as chemical intermediate for the synthesis of a cyclopropyl derivative.

20 However, in said document there is no mention, nor suggestion, of the olfactory properties of the compounds of the present invention.

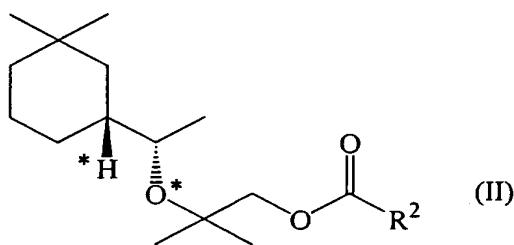
Description of the invention

25 Surprisingly, we have now established that a compound of formula



in the form of any of its isomers or a mixture thereof, and in which R¹ represents a linear, branched or cyclic C₂ to C₆ alkenyl, alkadienyl or aromatic group; possesses surprising odor properties, of the musky and green type, which have been found to be particularly useful and appreciated for the preparation of perfumes, perfuming compositions and perfumed products.

5 According to a particular embodiment of the invention, a compound of formula:



10 in the form of any of its isomers or a mixture thereof, and in which R² represents a linear, branched C₂ or C₃ 1-alkenyl group, and the hydrogen and oxygen atom marked with an asterisk are in a trans configuration, is preferred.

15 In particular, (1S,1'R)-2-[1-(3',3'-dimethyl-1'-cyclohexyl)ethoxy]-2-methylpropyl 2-propenoate possesses a powerful and persistent musky and green odor which is very original. More precisely the odor of said compound possesses at the same time a musky-ambrette and a fresh green character, together with a Galbanum and green pear undernote.

20 To the best of our knowledge, the combination of a musky and green character in the odor of a single compound has never been reported in the prior art. Moreover, the odor of said compounds has been found to be also very diffusive, a property which is quite rare for a compound possessing a musky note.

Given that the invention's compounds are valuable perfuming ingredients, the invention concerns also the use of a compound of formula (I) as perfuming ingredients. In other words it concerns a method to confer, enhance, improve or modify the odor properties of a perfuming composition or of a perfumed article, which method comprises adding to said composition or article an effective amount of at least a compound of formula (I). By "use of a compound of formula (I)" it has to be understood here also the use of the compound (I) in any of its forms which can be advantageously employed in perfumery as active ingredients.

Said forms are also an object of the present invention.

In an embodiment of the invention, one of said forms, which can be advantageously employed as perfuming ingredient, is a composition of matter consisting of at least a compound of formula (I) and at least one perfumery carrier. By "perfumery carrier" we mean here one or more materials which are able to be admixed with an invention's compound without significantly altering its organoleptic properties, e.g. materials which are neutral from a perfumery point of view. Said carrier may be a liquid.

As liquid carrier one may cite, as non-limiting examples, an emulsifying system, i.e. a solvent and a surfactant system, or a solvent commonly used in perfumery. As examples of solvents commonly used in perfumery, generally speaking, one can cite compounds such as dipropylene glycol, diethyl phthalate, isopropyl myristate, benzyl benzoate, 2-(2-ethoxyethoxy)-1-ethanol or ethyl citrate, which are the most commonly used.

In another embodiment of the invention, a suitable form of the invention's compound is a composition of matter comprising a compound of formula (I) and a perfume base. In other words the compound (I) is in the form of a perfuming composition. It is understood that the perfuming ingredients are present in a perfuming effective amount.

Generally speaking, by "perfume base" we mean here a composition comprising at least one perfuming co-ingredient and possibly one or more solvents or adjuvants commonly used in the perfume industry.

Said perfuming co-ingredients are not of the formula (I) and may be in any of their forms. Moreover, by "perfuming co-ingredient" it is also meant here a compound, which is of current use in perfumery industry, i.e. a compound which is used as ingredient in perfuming preparation or composition in order to impart an hedonic effect. In other words such a co-ingredient, to be considered as being a perfuming one, must be recognized by a person skilled in the art as being able to impart or modify in a positive or pleasant way the odor of a composition, and not just as having an odor. Its is therefore understood here that, unless otherwise indicated or described, any mixture resulting directly from a chemical synthesis in which the compound of the invention is involved as a starting

intermediate or as an end-product is not a perfuming composition according to the invention.

The nature and type of the perfuming co-ingredients present in the base do not warrant a more detailed description here, which in any case would not be exhaustive, the 5 skilled person being able to select them on the basis of its general knowledge and according to intended use or application and the desired organoleptic effect. In general terms, these perfuming co-ingredients belong to chemical classes as varied as alcohols, aldehydes, ketones, esters, ethers, acetates, nitriles, terpene hydrocarbons, nitrogenous or sulphurous heterocyclic compounds and essential oils of natural or synthetic origin. Many 10 of these co-ingredients are in any case listed in reference texts such as the book by S. Arctander, Perfume and Flavor Chemicals, 1969, Montclair, New Jersey, USA, or its more recent versions, or in other works of a similar nature, as well as in the abundant patent literature in the field of perfumery.

Similarly, a detailed description of the nature and type of solvents commonly used 15 in perfuming bases cannot be exhaustive. A skilled person in the art is able to select them on the basis of the nature of the product to be perfumed. However, as non-limiting examples of solvents commonly used in perfumery bases, one can cite, in addition to the solvents mentioned above, also ethanol, water/ethanol mixtures, limonene or other terpenes, isoparaffins such as those known under the trademark Isopar[®] (origin: Exxon 20 Chemical) or glycol ethers and glycol ether esters such as those known under the trademark Dowanol[®] (origin: Dow Chemical Company).

The perfuming compositions according to the invention may be a simple mixture 25 of the various co-ingredients and solvents, or also in the form of a bi-phasic system such as an emulsion or microemulsion. Such systems are well known to a person skilled in the art.

As previously mentioned, a compound of formula (I) is a useful perfuming ingredient which can be advantageously used in all the fields of modern perfumery to positively impart or modify the odor of a consumer product into which said compound (I), in any of its form, is added. Consequently, a perfumed article comprising:

30 i) a compound of formula (I), or any of its forms above-mentioned; and
ii) a consumer product base,

is also an object of the present invention.

For the sake of clarity, it has to be mentioned that, by "consumer product base" we mean here an unperfumed consumer product, i.e. a consumable product such as a detergent or a perfume, or a part of said consumer product. Therefore, a perfumed article 5 according to the invention comprises at least a part of the whole formulation corresponding to a desired consumer product, e.g. a detergent, and an olfactive effective amount of at least an invention's compound, in any of its forms.

The nature and type of the constituents of the consumer product do not warrant a more detailed description here, which in any case would not be exhaustive, the skilled 10 person being able to select them on the basis of its general knowledge and according to the nature and the desired effect of said product.

Suitable unperfumed consumer products comprise solid or liquid detergents and fabric softeners as well as all the other articles common in perfumery, namely perfumes, colognes or after-shave lotions, perfumed soaps, shower or bath salts, mousses, oils or 15 gels, hygiene products or hair care products such as shampoos, body-care products, deodorants or antiperspirants, air fresheners and also cosmetic preparations. As detergents are intended applications such as detergent compositions or cleaning products for washing up or for cleaning various surfaces, e.g. intended for textile, dish or hard-surface treatment, whether they are intended for domestic or industrial use. Other perfumed 20 articles are fabric refreshers, ironing waters, papers, wipes or bleaches.

Some of the above-mentioned consumer product bases may represent an aggressive medium for the invention compounds, so that it may be necessary to protect the latter from premature decomposition, for example by encapsulation.

The proportions in which the compounds according to the invention can be 25 incorporated into the various aforementioned articles or compositions vary within a wide range of values. These values are dependent on the nature of the article to be perfumed and on the desired olfactive effect as well as the nature of the co-ingredients in a given base when the compounds according to the invention are mixed with perfuming co-ingredients, solvents or additives commonly used in the art.

For example, in the case of perfuming compositions, typical concentrations are in 30 the order of 1 % to 20 % by weight, or even more, of the compounds of the invention

based on the weight of the composition into which they are incorporated. Concentrations lower than these, such as in the order of 0.05% to 1% by weight, can be used when these compounds are incorporated into perfumed articles.

The invention will now be described in further detail by way of the following examples, wherein the abbreviations have the usual meaning in the art, the temperatures are indicated in degrees centigrade (°C) ; the NMR spectral data were recorded in CDCl_3 (if not stated otherwise) with a 360 MHz machine for ^1H and 90.5 MHz for ^{13}C , the chemical displacement δ are indicated in ppm with respect to the TMS as standard, the coupling constants J are expressed in Hz.

10

Example 1

Synthesis of (1S,1'R)-2-[1-(3',3'-dimethyl-1'-cyclohexyl)ethoxy]-2-methylpropyl 2-propenoate

15

(1S,1'R)-2-[1-(3',3'-dimethyl-1'-cyclohexyl)ethoxy]-2-methyl-1-propanol, having an enantiomeric purity of ~85% and a diastereoisomeric purity of ~80%, was obtained according to the method described in EP 472966 and using a starting compound having a comparable enantiomeric and diastereoisomeric purity.

20 To a suspension of NaH (0.368mole) in 1000 ml of dry THF at 0° (ice-bath) were added dropwise 70.0g of (1S,1'R)-2-[1-(3',3'-dimethyl-1'-cyclohexyl)ethoxy]-2-methyl-1- propanol (0.306mole, $[\alpha]_D^{20} = +10.6^\circ$ (1.7%, CHCl_3), diastereoisomeric purity ~80%). After the end of the hydrogen evolution, 33.3g (0.368mole) of acryloyl chloride were added dropwise and the mixture thus obtained was stirred for 90 minutes at room 25 temperature, then for 48 hours at reflux. After cooling at 0° and the addition of 350ml of water, the reaction mixture was poured into 250ml of 1M HCl, extracted twice with 600ml of ether, washed once with saturated NaHCO_3 , twice with brine, dried over Na_2SO_4 and concentrated under *vacuum*. A distillation on residue ($E_b_{(0.05\text{mbar})} = 73\text{-}96^\circ$) furnished 81.35g of 65% crude material which were purified by flash chromatography over SiO_2 30 (eluent: pentane/ether= 96:4). The more pure fractions were mixed together, concentrated

under *vacuum* and distilled (bulb to bulb, 0.06mbar, oven = 125°) to give 64.4g (yield=74%) of a mixture of diastereoisomers containing ~81% of the title compound.

$[\alpha]_{20}^D = + 5.6^\circ$ (1.3%, CHCl_3)

5 $^1\text{H-NMR}$: 0.86 (s, 3H), 0.89 (s, 3H), 1.06 (d, $J = 6$, 3H), 1.21 (s, 6H), 1.27-1.72 (m, 7H),
 3.38 (q, $J = 6$, 1H), 4.03 (s, 2H), 5.84 (d, $J = 10.3$, 1H), 6.15 (dd, $J_1 = 10.3$, $J_2 = 17.4$, 1H), 6.43 (d, $J = 17.4$, 1H).
 $^{13}\text{C-NMR}$: 19.7(q), 22.3(t), 23.8(q), 24.2(q), 24.7(q), 28.3(t), 30.7(s), 33.7(q), 39.4(t),
 40.4(d), 42.3(t), 70.5(t), 71.9(d), 73.8(s), 128.6(d), 130.7(t), 166.1(s).

10

Example 2

Preparation of a perfuming composition

15 A perfuming composition of the "floral-musky" type for a powder detergent was prepared by admixing the following ingredients :

	<u>Ingredient</u>	<u>Parts by weight</u>
	Hexylcinnamic aldehyde	280
20	2-Methyldecanal	20
	9-Undecenal	20
	Verdyl acetate	80
	Coumarine	20
	Allyl cyclohexylpropionate	20
25	2-(4-Isopropylbenzyl)-2-methylpropanal	20
	Dihydromyrcenol	240
	Doremox ^{® 1)}	10
	Iralia ^{® 2)} Total	20
	Patchouli	10
30	Verdyl propionate	80
	Amyl salicilate	20
	Verdox ^{® 3)}	60

Yara Yara	20
2,4-Dimethyl-3-cyclohexen-1-carbaldehyde	10
Lavandin Grosso	<u>10</u>
Total	940

5

- 1) Tetrahydro-4-methyl-2-phenyl-2H-pyran; origin : Firmenich SA, Geneva, Switzerland
- 2) Mixture of methylionone isomers; origin : Firmenich SA, Geneva, Switzerland
- 3) 2-Tert-butyl-1-cyclohexyl acetate; origin : International Flavors & Fragrances, USA

10 The addition of 60 parts by weight of (1S,1'R)-2-[1-(3',3'-dimethyl-1'-cyclohexyl)ethoxy]-2-methylpropyl 2-propenoate to the above-described perfuming composition imparted to the latter a powerful musky aspect, which is also smooth and fresh, together with a green, pear's peel connotation and a Galbanum undernote.

15

Example 3

Preparation of a perfuming composition

20 A perfuming composition of the "floral-musky" type for a softener was prepared by admixing the following ingredients :

	<u>Ingredient</u>	<u>Parts by weight</u>
	Verdyl acetate	50
	Cinnamic alcohol	20
25	Hexylcinnamic aldehyde	250
	Benzophenone	5
	Benzylacetone	30
	Citronellol	60
	Allyl cyclohexylpropionate	5
30	Dihydromyrcenol	60
	Doremox® ¹⁾	5
	Iralia® ²⁾ Total	90

	2,4,6-Trimethyl-4-phenyl-1,3-dioxane	10
	Hedione ^{®3)}	30
	10%* Indomethylene	20
	10%* Neobuteneone ^{®4)}	20
5	10%* Trans-1-(2,2,6-trimethyl-1-cyclohexyl)-3-hexanol	15
	Rose oxide	30
	Phenylhexanol	80
	Hexyl salicilate	60
	Vertofix Coeur ^{®5)}	<u>60</u>
10	Total	900

*in dipropylene glycol

- 1) Tetrahydro-4-methyl-2-phenyl-2H-pyran; origin : Firmenich SA, Geneva, Switzerland
- 2) Mixture of methylionone isomers; origin : Firmenich SA, Geneva, Switzerland
- 15 3) Methyl dihydrojasmonate; origin : Firmenich SA, Geneva, Switzerland
- 4) 1-(5,5-Dimethyl-1-cyclohexen-1-yl)-4-penten-1-one; origin : Firmenich SA, Geneva, Switzerland
- 5) Origin : International Flavors & Fragrances, USA

20 The addition of 100 parts by weight of (1S,1'R)-2-[1-(3',3'-dimethyl-1'-cyclohexyl)ethoxy]-2-methylpropyl 2-propenoate to the above-described composition imparted to the latter a powerful musky aspect, which is well perceivable on the bottle as well as on the linen. Moreover, the presence of the invention's compound, in the above-mentioned composition, exalted the fresh green character which was originally imparted by the

25 Neobuteneone[®] and Doremox[®].

Example 4

Preparation of a perfuming composition

30 A woman's eau de toilette of the "ozonic-floral" type for a softener was prepared by admixing the following ingredients :

	<u>Ingredient</u>	<u>Parts by weight</u>
	1%* Phenylacetic aldehyde	20
	Beta Ionone	50
	Citronellol	50
5	γ -n-Decalactone	5
	Eugenol	10
	Hedione [®] HC ¹⁾	150
	3-(1,3-Benzodioxol-5-yl)-2-methylpropanal	50
	10%* Indol	15
10	Iso E super ^{® 2)}	200
	Lilial ^{® 3)}	150
	Linalool	100
	Myroxide ^{® 4)}	40
	10%* Neobuteneone ^{® 5)}	20
15	10%* 2,6-Dimethyl-5-heptanal	20
	7-Tert-butyl-2H,4H-1,5-benzodioxepin-3-one	10
	Feuillage vert base 47062 ⁶⁾	<u>10</u>
	Total	900

20 *in dipropylene glycol

1) Methyl dihydrojasmonate; origin : Firmenich SA, Geneva, Switzerland

2) 1-(Octahydro-2,3,8,8-tetramethyl-2-naphthalenyl)-1-ethanone; origin : International Flavors & Fragrances, USA

3) 3-(4-Tert-butylphenyl)-2-methylpropanal; origin : Givaudan-Roure SA, Vernier, Switzerland

4) 6,7-Epoxy-3,7-dimethyl-1,3-octadiene; origin : Firmenich SA, Geneva, Switzerland

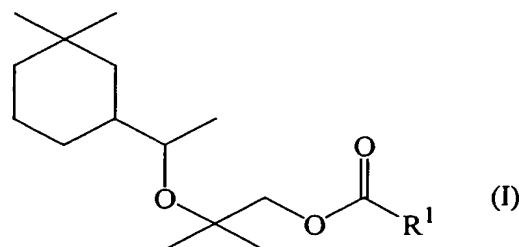
5) 1-(5,5-Dimethyl-1-cyclohexen-1-yl)-4-penten-1-one; origin : Firmenich SA, Geneva, Switzerland

6) Compounded perfumery base; origin : Firmenich SA, Geneva, Switzerland

The addition of 100 parts by weight of (1S,1'R)-2-[1-(3',3'-dimethyl-1'-cyclohexyl)ethoxy]-2-methylpropyl 2-propenoate to the above-described eau de toilette imparted to the latter a transparent, crystalline and fresh musky character. The eau de toilette thus obtained acquired more volume and impact, and become more natural and 5 fine perfumery. The addition of the invention's compound also rounded the Galbanum note, provided by the Neobuteneone®, by providing to the latter a sparkling and "mouth-watering" note of the powdery-fruity-pear's peel type.

Claims

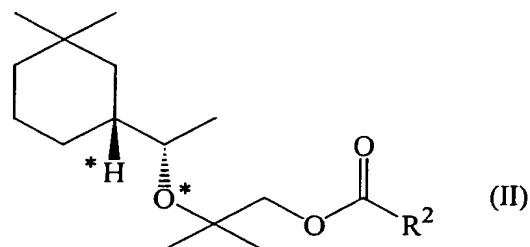
1. A compound of formula



in the form of any of its isomers or a mixture thereof, and in which R¹ represents a linear, branched or cyclic C₂ to C₆ alkenyl, alkadienyl or aromatic group.

10

2. As a compound according to claim 1, of formula



15

in the form of any of its isomers or a mixture thereof, and in which R² represents a linear, branched C₂ or C₃ 1-alkenyl group, and the hydrogen and oxygen atom marked with an asterisk are in a trans configuration.

3. As a compound according to claim 2, (1S,1'R)-2-[1-(3',3'-dimethyl-1'-cyclohexyl)ethoxy]-2-methylpropyl 2-propenoate

20

4. A composition of matter consisting of at least a compound of formula (I), as defined in any one of claims 1 to 3, and at least one perfumery carrier.

5. A composition of matter comprising a compound of formula (I), as defined in any one of claims 1 to 3, and a perfume base.

6. A perfumed article comprising:

5 i) a compound of formula (I), as defined in any one of claims 1 to 3, or a composition as defined in claim 4 or 5; and

ii) a consumer product base.

7. A perfumed article according to claim 6, characterized in that the consumer product base is a solid or liquid detergent, a fabric softener, a perfume, a cologne or after-shave lotion, a perfumed soap, a shower or bath salt, mousse, oil or gel, a hygiene product, a hair care product, a shampoo, a body-care product, a deodorant or antiperspirant, an air freshener, a cosmetic preparation, a fabric refresher, an ironing water, a paper, a wipe or a bleach.

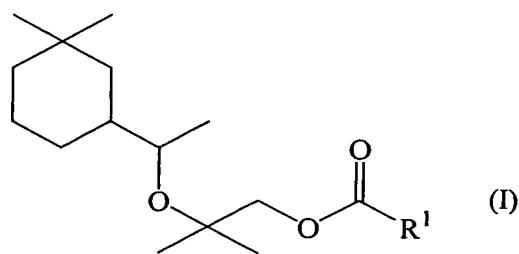
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8. Use as perfuming ingredient of a compound of formula (I), as defined in any one of claims 1 to 3, or of a composition as defined in claim 4 or 5.

Abstract

The present invention relates to the field of perfumery, and more precisely to a compound of formula (I)

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wherein R¹ represents an unsaturated hydrocarbon radical.

The present invention concerns also the use of said compound in the perfumery industry as well as the compositions or articles associated with said compound.

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